Effects of High-Pressure Treatment on Poplar Wood: Density Profile, Mechanical Properties, Strength Potential Index, and Microstructure

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The density profile, mechanical properties, strength potential index, and microstructure changes of hybrid poplar were investigated before and after high-pressure (HP) treatments. The results of density profile indicated that a high uniform density distribution was developed inside the pressurized wood samples. The mechanical properties results showed that the HP treatments significantly increased ($P < 0.05$) the modulus of elasticity (MOE), the modulus of rupture (MOR), and the Brinell hardness (BH) of the densified wood at selected conditions. Of all the wood samples, the compressed wood at 150 MPa condition possessed the highest density and strength properties. Considering the variation in strength properties along with density, it can be concluded that the compression destruction degree of HP treatment was comparable with that caused by optimized thermal compression technique based on the strength potential index results. The integrity of wood cells presented in scanning electron microscopy results demonstrated the compression of wood cell wall achieved by HP treatment without causing any fractures, which further indicated that HP treatment is a less destructive compression technology. Based on this research, HP treatment has great potential to be applied in wood densification for commercial use.

Keywords: High-pressure treatment; Hybrid poplar; Density profile; Mechanical property; Strength potential index; Microstructure

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INTRODUCTION

The strength properties of wood are positively related with its density. As a renewable and environmentally friendly material, high-density wood is widely used in daily life (*e.g.*, the construction, furniture, and floor industry). However, the supply of such wood is far from satisfying the growing demand of the market, mostly because of its long growth period. To solve this problem, fast-growing plantation forests could be used as a fungible resource, as they have not been effectively exploited because of the poor mechanical properties in relation to its low density. To make full use of the fast-growing forest resource, two main modification methods to improve wood density and mechanical properties have been developed. The first solution is impregnation method that involves filling wood cell cavities with fluid substances (Devi *et al.* 2003; Sun *et al.* 2016). The other way is to densify wood through compression by reducing its void volumes without adding any chemicals (Navi and Heger 2004; Welzbacher *et al.* 2008; Fang *et al.* 2012;

Laine *et al.* 2016). Since the impregnation method can cause damage to ecological characteristics of natural wood, making the material into one containing potentially harmful residues that can be harmful to both the environment and human health, compression technologies are more widely used in wood densification for furniture use.

The wood compression technology originally emerged in the beginning of the 20th century. At the early stage, wood samples were directly compressed to desired thickness in the radial direction using heated metal plates. With better understanding of the composition and natural characteristics of wood, unidirectional static compression with softening pre-treatment and cooling post-treatment was used to reduce the defects caused by compression processing. This technique resulted in an improvement of the quality of densified wood products. During the past few decades, thermo-hydro-mechanical (THM) compression, thermo-mechanical (TH) compression, viscoelastic thermal compression (VTC), and sandwich compression have been developed (Navi and Heger 2004; Welzbacher *et al.* 2008; Kutnar *et al.* 2009; Fang *et al.* 2012; Gao *et al.* 2016; Laine *et al.* 2016). Navi and Girardet (2000) reported that the mechanical properties of spruce, pine, and beech wood were significantly enhanced by THM treatment, particularly in the shear strength, with the increase rate of more than tenfold. Kutnar *et al.* (2008) found that the modulus of rupture (MOR) and modulus of elasticity (MOE) of VTC wood with a 132% degree of densification were 103% and 129% greater than those of non-densified hybrid poplar wood. However, these hot-pressing densification methods have some fatal drawbacks, such as complicated procedures and long treatment time, which result in low productivity and slow industrialization of these technologies.

Recently, several novel wood compression methods have been studied. Bucur *et al.* (2000) treated small pieces of spruce (at 5 MPa) and cherry (at 19 MPa) with hydrostatic pressure in a hermetically sealed cylindrical vessel. They found that the average density was improved by 26% for spruce and 46% for cherry. Moreover, the densified woods became less heterogeneous and less anisotropic than before. In another study, Blomberg and Persson (2004) studied the effects of semi-isostatic compression on properties of Scots pine in a Quintus press that can yield pressures of up to 140 MPa. Based on their findings, they concluded that the application of pressure in all directions contributed to the prevention of compression defects (*e.g.*, fracture, fragmentation, spreading, checking). Li *et al.* (2016) applied high-pressure (HP) treatments (*i.e.*, purely isostatic compression) to small Chinese fir logs to modify wood properties. Results showed that pressurized woods possessed better mechanical properties and stability at a moderate relative humidity (RH). Above all, these results based on previous studies suggest that purely isostatic compression is a less destructive wood compression technique compared with traditional compression technologies. In conventional wood production industry, logs are generally sawn into wood boards to speed up the drying process. To our best knowledge, the effects of HP treatment on the density distribution, mechanical properties, and microstructure of wood boards have not been studied yet.

The objective of this study was to investigate the effects of HP treatment on the density and mechanical properties of hybrid poplar boards. The detrimental compression effects on the enhanced mechanical performances were compared between HP densification and other thermal compression methods through the strength potential index. In addition, a scanning electron microscopy analysis was conducted to examine the microscopic changes in HP-compressed wood samples and to diagnose how detrimental the HP densification has been to the cell walls.

EXPERIMENTAL

Wood Samples

Hybrid poplar (*Populus* × *euramericana* 'Neva') was provided by a plantation forest in Henan Province, China. Plain boards (850 mm × 300 mm × 35 mm) were sawn from poplar logs and were air-dried in a laboratory for several months. After drying, all dried boards were cut into rectangular wood specimens with the size of 180 mm (longitudinal) × 80 mm (tangential) × 30 mm (radial). Prior to HP treatment, all prepared specimens were conditioned under 20 °C and 60% RH until equilibrium was reached. To ensure the consistency of wood samples, all specimens were carefully selected with similar weight (200 ± 5 g) and free of macroscopic defects (*e.g.*, knots, cracks, and splits).

High-Pressure Treatment

HP treatment was conducted using laboratory-scale HP equipment (UHPF-750, Kefa, Baotou, China), with a maximum volume of 5 L and 600 MPa pressure limit. In this study, purified water was used as pressure transmitting medium. The initial temperature of the transmitting medium in the pressure vessel was kept at 25 °C. The temperature rise caused by adiabatic compression was expected to be 3 °C for every 100-MPa pressure rise. Considering the low applied pressure and short pressure holding time (30 s), the temperature of pressure transmitting medium and wood samples during HP processing was considered to be constant (25 °C). The intensifier used a stepwise ladder-like process to generate pressure. The average pressure build-up rate was approximately 100 MPa/min, and the depressurization time was less than 5 s. Firstly, the prepared wood specimens were clamped with iron plates on both tangential surfaces and vacuum-packaged with polythene bags. The HP treatment was conducted at 50, 100, 150, and 200 MPa for a 30 s. Each combination was carried out with five replicates. After treatment, three straight lines were drawn on each cross-section of the densified wood specimens to determine the thickness changes in the compression direction. In this study, the dimensions of specimens were measured using a digital micrometer (FH-150, Xifeng, China) with an accuracy of 0.03 mm and repeatability of 0.01 mm. Three replicates were tested for each measurement. The compression ratio (CR) of the thickness direction was measured using Eq. 1, where T_o and T_c are the thickness of specimens in air-dry conditions before and immediately after densification, respectively.

$$\text{Compression ratio (\%)} = \frac{T_o - T_c}{T_o} \times 100 \tag{1}$$

Thickness Swelling Test

After HP treatment, all samples were conditioned at 20 °C and 60% RH until the thickness became stable. Then, the thickness of the compressed samples was measured at 1, 24, 48, 72, 96, 120, 144, and 168 h after the treatment. After the swelling test, the specimens were still stored at 20 °C and 60% RH for subsequent experiment. The thickness swelling (TS) was calculated using Eq. 2, where T_d is the thickness in air-dry conditions during the swelling test.

$$\text{Thickness swelling (\%)} = \frac{T_d - T_c}{T_c} \times 100 \tag{2}$$

Density Profile Measurement

For the density profile measurement, sections sized 50 mm × 50 mm were cut from the non-densified and densified wood specimens, while the thickness remained constant at a compressed state. The density profile of the sections was measured using a cross-sectional X-ray densitometer (DENSE-LAB X, EWS, Germany) with an interval of 0.2 mm through the thickness. Three replicates were tested for each group.

Mechanical Tests

All mechanical tests in this study were conducted using a universal mechanical testing machine (CMT4204, MTS, China) equipped with a 20-kN load cell. Both the modulus of rupture (MOR) and the modulus of elasticity (MOE) were measured by a three-point static bending test with reference to GB/T 1936 (2009). The specimens sized 150 mm × 12 mm × 5 mm were used because the laboratory-scale HP equipment only produced small densified wood, and the span of supports was adjusted to 100 mm. The Brinell hardness (BH) was determined in accordance with GB/T 1941 (2009), and the test samples were sized 70 mm × 50 mm × 10 mm. In this method, a hemispherical ball with a radius of 5.64 mm penetrated to a depth of 2.82 mm on the tangential surface of specimens at 3 mm/min. The measurements were taken on six replicates for hardness and 23 replicates for MOE and MOE.

Determination of Strength Potential Index

Further analysis of the effects of densification was determined by calculating the ratio between the performance prior to and after densification. The density index (ρ_d/ρ_o) and the strength index (f_d/f_o) reflect the increase in density and strength. The strength potential index proposed by Blomberg *et al*. (2005) was used to estimate how much the strength of densified wood increases relative to what could be expected for non-densified wood from the increase in density, and was measured according to Eq. 3. It should be noted that the constant "b" given in Table 1 is from the function $\ln f = \ln a + b \ln \rho$, which indicates the relationship between the density and strength.

$$\ln f = \ln a + b \ln \rho \tag{3}$$

Table 1. Constants *a* and *b* Describe the Relationship between Density (*ρ*) and Strength (*f*)

Strength Property	ln *a*	*b*	Density Range (kg/m^3)	Source
Modulus of rupture (MOR)	-3.46	1.25	300-1000	Bodig and Jayne (1982)
Modulus of elasticity (MOE)	-5.88	1	300-1000	Liu and Zhao (2004)
Brinell hardness (HB_{tang})	-12.9	2.14	200-1000	Kollmann and Côté (1984)

Coefficients *a* and *b* given in the literature for functions according to the model: $\ln f = \ln a + b \ln \rho$

Scanning Electron Microscopy

Thin sections were prepared by a sliding microtome from air-dried specimens. The obtained sections were adhered to a silver specimen holder with a double-sided conductive carbon tape and then were coated with a thin film of gold (10 nm) in a vacuum evaporator using ion sputtering (E-1010, HITACHI, Japan). The surfaces of the wood samples were investigated by a scanning electron microscope (TM3000, HITACHI, Japan) at 15 kV to analyze the microstructure changes caused by densification.

Statistical Analysis

One-way analysis of variance (One-way ANOVA) was conducted using SPSS (version 20.0, IBM, USA) to analyze statistical differences among all tested wood specimens, Duncan's multiple range test was applied ($P < 0.05$). Different lowercase letters indicated significant differences, and the significance level was marked in alphabetical order from maximum to minimum. Results were presented as the mean values with standard deviations. Graphic presentations were plotted using Origin software (Version 8.0, OriginLab, USA) and MATLAB (Version 2013b, MathWorks, USA).

RESULTS AND DISCUSSION

Compression Ratio and Thickness Swelling

Results of thickness, compression ratio (CR), and thickness swelling (TS) of wood samples treated under various conditions are given in Table 2. An obvious change caused by HP treatment was observed in the specimen thickness. The reduction in the thickness of the compressed wood was determined by the compression ratio. As shown, most of the total deformations (45% CR) occurred below 50 MPa. Only small compression deformations (approximately 6.5% CR) happened with the pressure 50 to 100 MPa, and no remarkable change was found after 100 MPa. Similar results were found in earlier studies by Blomberg and Persson (2004). Ahmed *et al.* (2013) reported that the compression deformation was affected by anatomical features of the wood, such as the cell wall thickness and cell cavity size. The thinner the cell wall is, or the bigger the cell lumen of wood is, the more the cell is deformed. In this research, the vessels of hybrid poplar, with a relatively thin cell wall and large cell lumen, were easily collapsed, even at low pressure. That’s why most of the deformations occurred below 50 MPa.

Table 2. Thickness, Density, Compression Ratio, and Thickness Swelling of Treated Poplar Wood

Pressure (MPa)	Thickness (mm)	Density (kg/m^3)	Compression Ratio (%)	Thickness Swelling (%)
Control	29.25±0.56 a	484.15±6.82 d	-	-
50	17.19±1.06 b	826.18±10.81 c	45.04±2.46 b	7.79±2.44 a
100	15.20±0.38 c	947.97±46.08 b	51.54±0.59 a	8.17±2.67 a
150	14.49±0.48 c	999.98±25.55 a	53.35±1.09 a	7.10±2.38 a
200	14.84±0.40 c	971.00±11.91 ab	52.46±1.84 a	7.72±3.39 a

All values are expressed as means ± SD. Sample means with different lowercase letters in the same column are significantly different ($P < 0.05$). Note: the average swelling was calculated based on the stable state (ranging from day 4 to day 7)

Figure 1 shows that the TS of the densified woods changed with storage time. Similar trends were observed for all tested specimens. During the first hour after the test, the thickness of the densified wood rapidly increased by 3.5%. Continuous increase trends were observed in all groups until 72 h, and the TS ceased after 96 h. Wood is a type of viscoelastic material, the cell deformations caused by the compression can result in internal stresses stored in the microfibrils and matrix of the wood, which is the reason for the spring-back of densified wood. As shown in Table 2, the final TSs, on average, were less than 10%, which was similar to hot-pressing compression at the same CR level (Welzbacher *et al.* 2008; Laine *et al.* 2016). In addition, there was no significant difference (P > 0.05) in the final TS among all HP-densified specimens. All the subsequent experiments were conducted after 168 h of the swelling test.

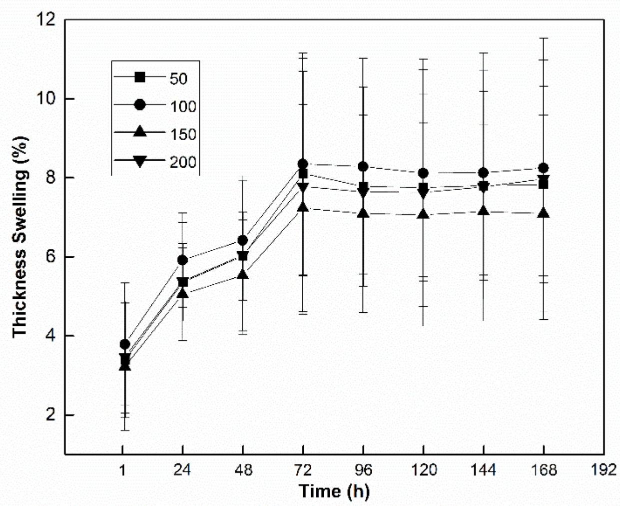

Fig. 1. Average TS of HP-compressed wood samples as a function of time during the swelling test

Effects of HP on Density

The density profile, which characterizes the density through the wood thickness, is an important attribute because it affects both the physical and mechanical properties of wood. As shown in Fig. 2, the non-densified wood exhibited almost uniform density with respect to the thickness direction. This was expected, since hybrid poplar is a rather homogeneous hardwood (Standfest *et al.* 2013). Additionally, the tested samples had no defects (*e.g.*, knots), which also contributed to developing a constant density distribution. Moreover, the density profile results verified that the density profile of all HP-compressed specimens differed from that of previous hot-pressing compression methods (Kutnar *et al.* 2009; Gao *et al.* 2016). The N-shaped density gradient across the thickness showed the lower density region on both sides and obvious higher plateau region in the middle. This phenomenon was observed in all HP-treated samples, which was most probably caused by the combination effect of HP treatment and spring-back. Bucur *et al.* (2000) reported that shearing deformation from hydrostatic pressure caused some overlaps between latewood and earlywood, which led to a decrease in the heterogeneity of the density for both spruce and cherry. Blomberg and Persson (2004) also reported that a softer structure was more easily densified than the harder one under semi-isostatic compression, which made the wood density more homogenous. A decline in the density profile was observed in the surface regions of the HP-treated wood. This was most probably caused by the spring-back, as discussed before. Additionally, Antikainen *et al.* (2014) stated that the low density recorded at the surfaces of densified wood might be a measurement error because of its slightly uneven surfaces.

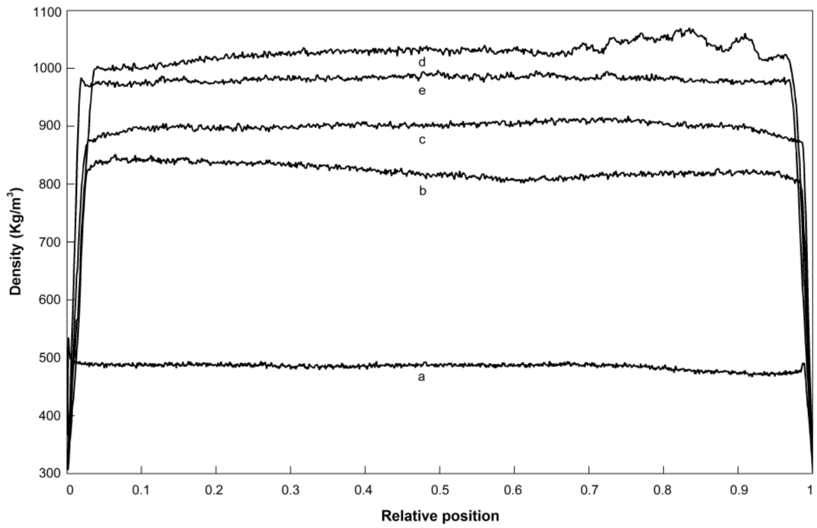

Fig. 2. Density profile of poplar wood samples treated under various conditions: a) control density profile; b) 50 MPa compressed density profile; c) 100 MPa compressed density profile; d) 150 MPa compressed density profile; e) 200 MPa compressed density profile

The average air-dry density of the control and densified wood was calculated based on an almost consistent density profile, and the results are shown in Table 2. The average density of non-densified control specimens was 484.15 kg/m^3, while remarkable increases of 71%, 96%, and 107% were observed at 50, 100, and 150 MPa, respectively. However, there was no obvious change ($P > 0.05$) in average density as the pressure increased to more than 150 MPa. Additionally, no significant variation of average density happened at pressure levels of 100 MPa and 200 MPa. It is a remarkable fact that the variation in density value became smaller when the pressure exceeded 100 MPa, and the growth percentages fluctuated around 100%. This abnormal trend may be a result of the reduction of void spaces in pressurized wood under the pressure conditions of more than 100 MPa. The increase of the wood density was realized by reducing its volume as the compression technique was based on the viscoelastic nature of the wood.

Effects of HP on Mechanical Properties

As shown in Fig. 3, the MOE dramatically increased with the increasing of pressure (ranging from 50 to 150 MPa), while a noticeable decline was observed when the pressure exceeded 150 MPa. The highest level of MOE was obtained at 150 MPa, with an increase of 162% in comparison with that of the control. A similar trend was observed in the modulus of rupture (MOR), as shown in Fig. 4. The HP treatment markedly improved the MOR of hybrid poplar wood, and there were no significant differences ($P > 0.05$) among tested specimens treated at 50, 100, and 200 MPa.

The density is an important property of wood since it is closely related with its mechanical properties. In the case of densified wood, the strength is generally in positive correlation with the density; thus, these change trends of mechanical properties were in accordance with that of its average density. It is an abnormal phenomenon that both MOE and MOR values of specimens treated at 200 MPa were lower than those at 150 MPa, though no significant difference of average density was observed between 150 MPa and 200 MPa treated specimens. This might have been caused by the destruction of the wood structure during HP treatment. Previous microscopic studies have shown that the compression process caused a substantial number of cracks and fractures in the cell wall of densified wood, especially at a high-compression ratio (Bucur *et al.* 2000; Blomberg *et al.* 2006; Ahmed *et al.* 2013; Budak *et al.* 2016). These cell deformation defects have a negative impact on mechanical properties of densified wood. The more damaged the wood structure is, the less strength the densified wood has at the same density level.

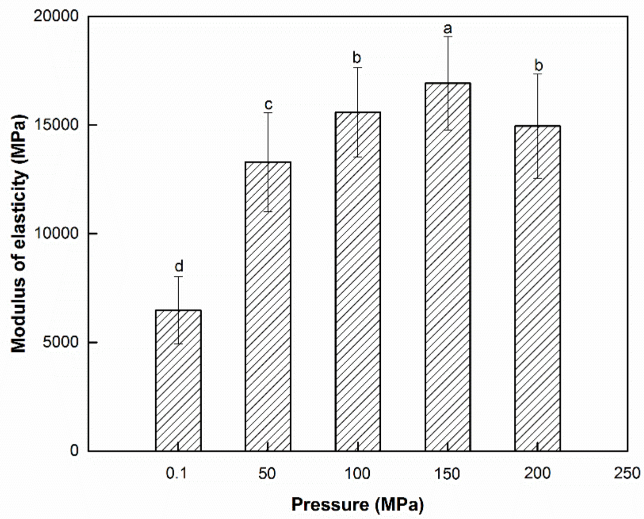

Fig. 3. Modulus of elasticity (MOE) of poplar wood samples treated under various conditions. The error bars indicate the standard deviation. Different letters above the columns indicate significant differences ($P < 0.05$).

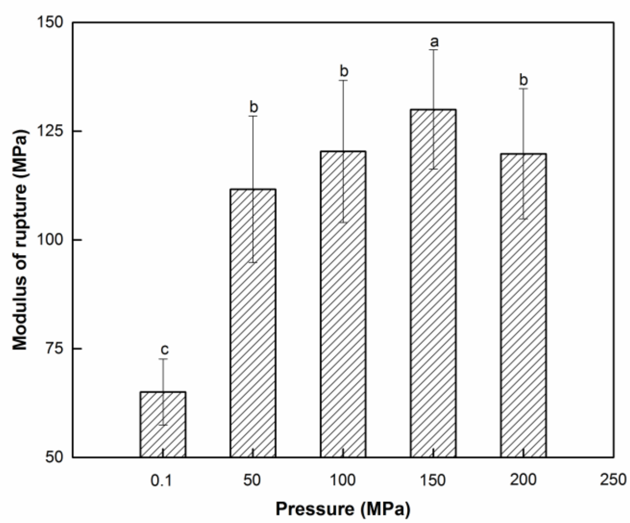

Fig. 4. Modulus of rupture (MOR) of poplar wood samples treated under various conditions. The error bars indicate the standard deviation. Different letters above the columns indicate significant differences ($P < 0.05$).

The Brinell hardness (BH) is a practical mechanical property to assess the resistance of wood. The BH results on the tangential surface of the control and HP-treated wood specimens are presented in Fig. 5. As can be seen, the BH value of non-densified specimens was only 1150.5 N; however, the values increased by 49%, 61%, 67%, and 55% after treatments at 50, 100, 150, and 200 MPa, respectively. Statistical analysis demonstrated that HP compression contributed to enhancing the hardness of poplar samples ($P < 0.05$). However, no significant change ($P > 0.05$) was found among samples compressed at different pressure levels. The increasing density and possible destruction of the wood structure also revealed the variation in the hardness with increased pressure.

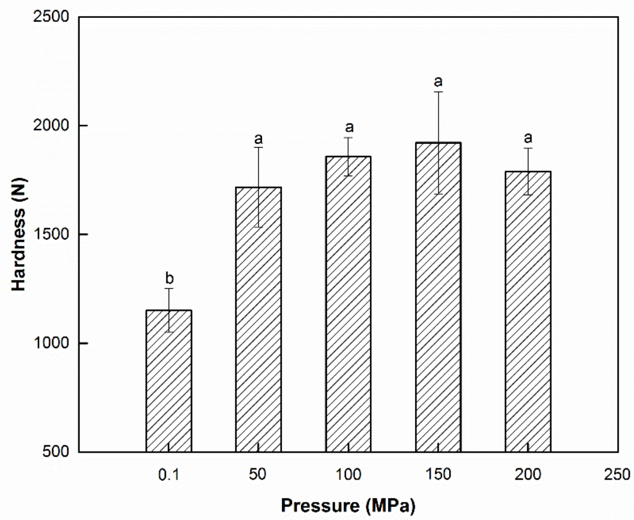

Fig. 5. Brinell hardness (BH) of poplar wood samples treated under various conditions. The error bars indicate the standard deviation. Different letters above the columns indicate significant differences ($P < 0.05$).

Altogether, these strength results indicated that HP treatment was analogous to traditional hot-pressing compression methods (*e.g.*, TH, THM, and VTC), which can greatly improve mechanical properties of low-density wood to substitute for harder species. Compared with traditional densification methods mentioned above (processing time ranges from 0.6 h to 3 h), the HP treatment used in this study had a relatively simpler compression procedures and had a shorter pressing time of 30 s, which can greatly promote the production efficiency.

Strength Potential Index Analysis

The compression of solid wood causes a general collapse in the cell structure, and possibly results in compression defects (*e.g.*, breaking, cracking). This may have a negative effect on wood's strength properties. The strength then usually increases less than the density in relative terms. In general, the more damage the wood structure suffers during densification, the worse its strength is at a given density after densification. Many researchers have reported the relationships between density and several strength properties among non-densified wood (Bodig and Jayne 1982; Kollmann and Côté 1984; Liu and Zhao 2004). Mostly, the strength to density relationship takes the form $f = a\rho^b$ or $\ln f = \ln a + b\ln\rho$, where the constants "a" and "b" differ among mechanical properties (Table 1). Considering the same strength property, the parameters "a" and "b" also vary much in different studies. This may be caused by the differences in wood species and individuals. Blomberg *et al.* (2005) reported that the mechanical properties of semi-isostatic densified woods varied with its density, and no obvious change was found in the strength to density relationship between densified wood and non-densified wood, except for the coefficient "*a*". Therefore, the relationship between density and mechanical properties can be used to diagnose how much damage the densification processing can cause to the cell walls. The strength of densified wood relative to what could be expected for non-densified wood of the same density is used to denote the "strength potential index".

The strength potential index (a_d/a_o) of the HP densification for different strength properties is given in Table 3. For MOR and MOE, the value of a_d/a_o was close to 1, which indicated that the HP-compressed wood was not negatively affected by compression in the axial direction. However, the a_d/a_o of hardness was very low, which showed that the densified wood was substantially weaker than what could be expected from its density in the compression direction. This appears to be associated with the deformation of rays during compression. The rays work as important reinforcements and affect the strength of non-densified wood in the radial direction. Additionally, Blomberg (2005) stated that 40% of immediate elastic strain accrued in the semi-isostatic densified Scots pine when pressure was released, which caused the wood to become rubbery. Therefore, the hardness of the densified wood was lower than theoretically assumed. It is noteworthy that the lowest a_d/a_o of all strength was found in 200 MPa treated specimens, which reflected that 200 MPa of treatment further pressed the wood samples and caused more compression defects on wood cells than other HP treatments.

Table 3. Ratios between Densified (d) and Non-Densified (0) Wood for Density (ρ), Strength (f), and Strength Potential Index ($a = f/\rho^b$)

Material	ρ_d/ρ_o	MOR		MOE		BH	
		f_d/f_o	a_d/a_o	f_d/f_o	a_d/a_o	f_d/f_o	a_d/a_o
Poplar, 50 MPa	1.71	1.72	0.88	2.05	1.20	1.49	0.48
Poplar, 100 MPa	1.96	1.85	0.80	2.41	1.23	1.61	0.38
Poplar, 150 MPa	2.07	2.00	0.81	2.62	1.27	1.67	0.35
Poplar, 200 MPa	2.01	1.84	0.77	2.31	1.15	1.55	0.35
Poplar, 40% CR[a]	1.60	1.42	0.79	1.52	0.95	1.64	0.60
Poplar, 50% CR[a]	1.82	1.45	0.69	1.70	0.93	1.85	0.51
Poplar, 160 °C [b]	1.97	2.25	0.96	2.29	1.16	3.34	0.78
Poplar, 180 °C [b]	2.04	2.25	0.92	2.33	1.14	2.72	0.59
Poplar, 200 °C [b]	2.10	2.21	0.87	2.81	1.34	2.79	0.57
Poplar, 220 °C [b]	2.06	2.05	0.83	3.00	1.45	2.35	0.50
Hybrid poplar 38% CR[c]	1.63	1.32	0.72	1.37	0.84	-	-
Hybrid poplar 50% CR[c]	1.98	1.66	0.71	1.84	0.93	-	-
Hybrid poplar 58% CR[c]	2.32	2.02	0.71	2.29	0.99	-	-

MOR (b=1.25), MOE (b=1), and BH (b=2.14) for HP-isostatic densification compared with other densification methods
[a] TM compression (Hu 2005)
[b] THM compression (Fang *et al.* 2012)
[c] VTC compression (Rutnar *et al.* 2008)

The strength potential index was also used as an indicator to compare the HP densification with other thermal densification methods (*e.g.*, TM, THM, and VTC) in the destructive level of wood cells. The a_d/a_o for traditional compression methods was calculated based on the data published in previous studies (Hu 2005; Rutnar *et al.* 2008; Fang *et al.* 2012). As shown in Table 3, the a_d/a_o for MOR and MOE were compared among different densification methods. The a_d/a_o at HP treatment tended to be higher than at TM and VTC compression, although the a_d/a_o value of HP-treated wood was slightly lower than the one of specimens treated with THM compression. This result confirmed that HP treatment enhanced strength properties more effectively than most of thermal compression technologies in the axial direction. However, the value of HP densification was slightly lower than that of all hot-pressing compression methods in terms of the a_d/a_o of hardness. The hardness property of wood is a reflection of the resistance of the wood surface; thus, it is closely related to the surface density. The densified wood treated with traditional densification usually has an uneven density distribution, *i.e.*, the surface density is greater than the internal density, while the HP-compressed wood possesses a rather uniform density distribution throughout its thickness. Therefore, the traditional densification method resulted in a higher hardness value compared with HP treatment, though the densified wood had a similar average density. The strength potential index results indicated that HP treatment might have been less destructive compared with other thermal compression methods.

It should be noted that small wood specimens compared in different densification methods were free of visible defects. The effects of the HP densification on deformation differed much between hard and soft structures. More deformation was found in soft structures, which might contribute to protecting the integrity of the wood cells and preventing densified wood from spreading and checking. HP treatment is probably the least detrimental wood compression technique, especially where knots and other possible defects of wood samples are concerned.

Scanning Electron Microscopy Analysis

Scanning electron microscopy (SEM) was performed to examine the anatomical changes caused by HP treatment. The SEM micrographs of cross-sections from the untreated and HP-treated specimens at different magnifications are shown in Fig. 6. Figure 6a shows the anatomical structure of the control, it is clear that hybrid poplar is a diffuse porous hardwood with thin-walled vessels and libriform fibers. There are no obvious differences between the earlywood and latewood, which explains why the control wood samples possessed relatively uniform density distribution.

According to the Figs. 6a to 6e, HP treatment distinctly changed the microstructure of hybrid poplar wood. Transects of the wood treated at 50 MPa showed that vessels were all collapsed and flattened, while the libriform fibers were slightly deformed. Microscopic examination showed that HP treatment at low pressure (50 MPa) made the thin-walled vessels collapse and consequently resulted in a high CR. Furthermore, higher pressure contributed to deforming the cell wall and reducing the cell lumen volume. After 100 MPa of treatment, only a small part of the cell cavity still remained open. The most severe deformation was observed at 150 MPa, where the vessel and fiber lumens were almost completely closed. This explained why the CR increased with increasing pressure up to 150 MPa. However, it seemed that the compression deformation recovered at 200 MPa. A slight increase in the volume of void spaces was found compared with samples treated at 150 MPa, which caused a slight decrease in CR and density at 200 MPa. As shown by the SEM images derived from a larger area (200 μm/100X magnification), the collapsed vessels and deformed cells were evenly distributed inside of the HP-compressed wood. Therefore, a rather uniform density profile was acquired.

As shown in microscopic images (20 μm/1500X magnification), all HP-treated wood specimens were free of obvious compression destruction (*e.g.*, fracture, rupture, fragmentation) in the cell wall, except the existence of some small cracks in the cell wall of 200 MPa-compressed wood specimens. The mechanical properties of the densified wood were determined by the destruction degree of the cell wall. In general, the less amount of destruction occurred in the cell wall, the better mechanical properties of wood (*e.g.*, MOE, MOR, and BH) will obtain. Thus, some small cracks of cell wall explained why the strength of HP-treated wood samples reduced at pressure level of 200 MPa.

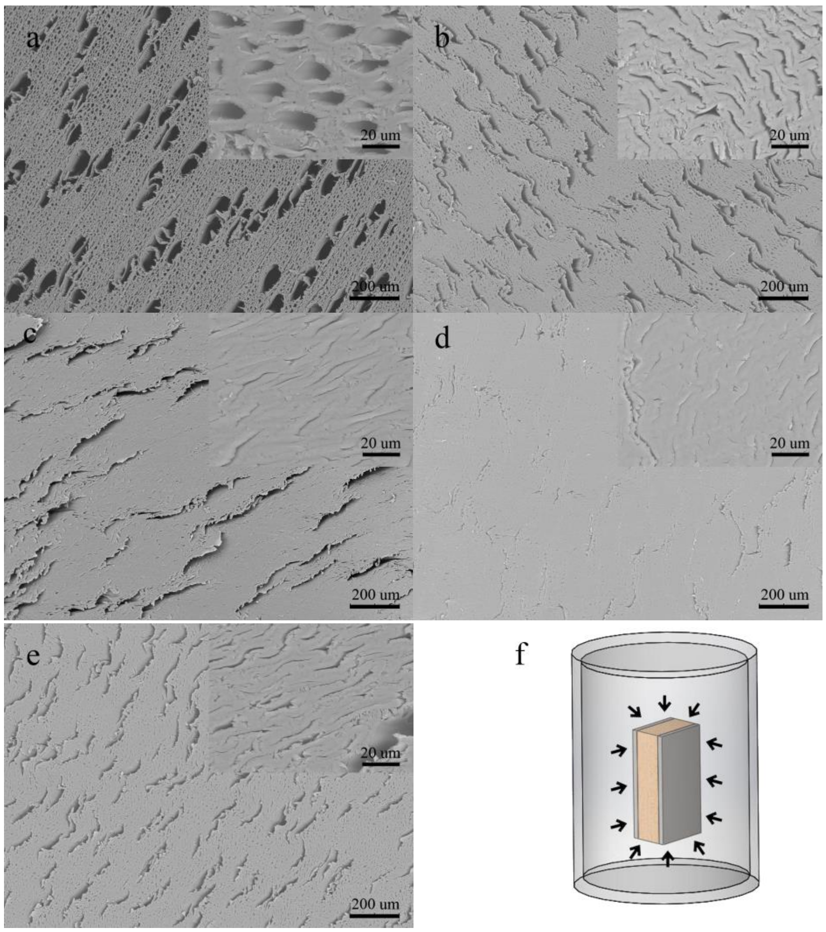

Fig. 6. SEM images of poplar wood samples treated under various conditions at different magnifications: a) control cross-section; b) 50 MPa compressed cross-section; c) 100 MPa compressed cross-section; d) 150 MPa compressed cross-section; e) 200 MPa compressed cross-section; f) shows the applied pressure direction of tested wood samples during HHP treatment

Additionally, the unbroken cell structure verified that HP treatment, as a less detrimental wood compression technique, could effectively enhance mechanical properties of low-density wood. Figure 6f describes the simulation chart of the HP processing applied to the tested wood. In this process, the water acted as pressure transmitting medium, which transferred pressure fast and evenly, and then the tested wood sample was compressed by the uniform pressure from all directions. The soft structure in the wood was more easily deformed by HP treatment compared with the hard structure, which reduced the compression defects and protected the integrity of wood cell.

Altogether, the morphology properties of the HP-compressed samples was consistent with previous studies concerning the morphology of optimized thermal compression (Navi and Girardet 2000; Kutuar *et al.* 2009; Ahmed *et al.* 2013; Laine *et al.* 2016). The results confirmed that HP processing is a less destructive compression method for wood.

CONCLUSIONS

1. HP treatment substantially enhanced the density and mechanical performances of the hybrid poplar boards at selected pressure levels. The results of density and mechanical properties indicated that 150 MPa may be the optimal pressure condition. In addition, a relatively uniform density distribution was obtained after high-pressure treatment at all researched conditions.
2. Results from strength potential index and SEM images demonstrated that HP treatment resulted in large deformations of the cell wall, without causing any fractures. As a consequence, HP treatment has been proved to be a less destructive wood compression technique compared with traditional thermal compression methods (*e.g.*, TH, THM, and VTC).
3. It can be inferred that HP treatment will be a good alternative technology in the wood compression industry. However, more studies are needed before putting this technique into commercial use.

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REFERENCES CITED

Ahmed, S. A., Moren, T., Hagman, O., Cloutier, A., Fang, C. H., and Elustondo, D. (2013). "Anatomical properties and process parameters affecting blister/blow formation in densified European aspen and downy birch sapwood boards by thermo-hygro-mechanical compression," *Journal of Materials Science* 48(24), 8571-8579. DOI: 10.1007/s10853-013-7679-9

Antikainen, T., Paajanen, O., Rautkari, L., Kutnar, A., Kamke, F. A., and Hughes, M. (2014). "Simultaneous drying and densification of silver birch (*Betula pendula* L.) veneers: Analysis of morphology, thickness swelling, and density profile," *Wood Science and Technology* 48(2), 325-336. DOI: 10.1007/s00226-013-0605-0

Blomberg, J. (2005). "Elastic strain at semi-isostatic compression of Scots pine (*Pinus sylvestris*)," *Journal of Wood Science* 51(4), 401-404. DOI: 10.1007/s10086-004-0666-7

Blomberg, J., and Persson, B. (2004). "Plastic deformation in small clear pieces of Scots pine (*Pinus sylvestris*) during densification with the CaLignum process," *Journal of Wood Science* 50(4), 307-314. DOI: 10.1007/s10086-003-0566-2

Blomberg, J., Persson, B., and Blomberg, A. (2005). "Effects of semi-isostatic densification of wood on the variation in strength properties with density," *Wood Science and Technology* 39(5), 339-350. DOI: 10.1007/s00226-005-0290-8

Blomberg, J., Persson, B., and Bexell, U. (2006). "Effects of semi-isostatic densification on anatomy and cellshape recovery on soaking," *Holzforschung* 60(3), 322-331. DOI: 10.1515/HF.2006.052

Bodig, J., and Jayne, B. A. (1982) *Mechanics of Wood and Wood Composites*, Van Nostrand Reinhold, New York, NY.

Bucur, V., Garros, S., and Barlow, C. Y. (2000). "The effect of hydrostatic pressure on physical properties and microstructure of spruce and cherry," *Holzforschung* 54(1), 83-92. DOI: 10.1515/HF.2000.013

Budak, M., Pelit, H. S., Nmez, A., and Korkmaz, M. (2016). "The effects of densification and heat post-treatment on hardness and morphological properties of wood materials," *BioResources* 11(3), 7822-7838. DOI: 10.15376/biores.11.3.7822-7838

Devi, R. R., Ali, I., and Maji, T. K. (2003). "Chemical modification of rubber wood with styrene in combination with a crosslinker: Effect on dimensional stability and strength property," *Bioresource Technology* 88(3), 185-188. DOI: 10.1016/S0960-8524(03)00003-8

Fang, C. H., Mariotti, N., Cloutier, A., Koubaa, A., and Blanchet, P. (2012). "Densification of wood veneers by compression combined with heat and steam," *European Journal of Wood and Wood Products* 70(1-3), 155-163. DOI: 10.1007/s00107-011-0524-4

Gao, Z. Q., Huang, R. F., Lu, J. X., Chen, Z. J., Guo, F., and Zhan, T. Y. (2016). "Sandwich compression of wood: control of creating density gradient on lumber thickness and properties of compressed wood," *Wood Science and Technology* 50(4), 833-844. DOI: 10.1007/s00226-016-0824-2

GB/T 1936.1 (2009) "Method of testing in bending strength of wood," Standardization Administration of China, Beijing, China.

GB/T 1936.2 (2009) "Method for determination of the modulus elasticity in static bending of wood," Standardization Administration of China, Beijing, China.

GB/T 1941 (2009) "Method of testing in hardness of wood," Standardization Administration of China, Beijing, China.

Hu, G. N. (2005) *Study on the Compression of Fast-Growing Wood of Poplar*, Master's Thesis, Fujian Agriculture and Forestry University, Fujiang, China.

Kollmann, F. F. P., and Côté, W. A. (1984) *Principles of Wood Science and Technology*, Springer, Berlin, Germany.

Kutnar, A., Kamke, F. A., and Sernek, M. (2008). "The mechanical properties of densified VTC wood relevant for structural composites," *Holz als Roh- und Werkstoff* 66(6), 439-446. DOI: 10.1007/s00107-008-0259-z

Kutnar, A., Kamke, F. A., and Sernek, M. (2009). "Density profile and morphology of viscoelastic thermal compressed wood," *Wood Science and Technology* 43(1-2), 57-68. DOI: 10.1007/s00226-008-0198-1

Laine, K., Segerholm, K., Wålinder, M., Rautkari, L., and Hughes, M. (2016). "Wood densification and thermal modification: Hardness, set-recovery and micromorphology," *Wood Science and Technology* 50(5), 883-894. DOI: 10.1007/s00226-016-0835-z

Li, H. H., Zhang, F. M., Ramaswamy, H. S., Zhu, S. M., and Yu, Y. (2016). "High-pressure treatment of Chinese fir wood: Effect on density, mechanical properties, humidity-related moisture migration, and dimensional stability," *BioResources* 11(4), 10497-10510. DOI: 10.15376/biores.11.4.10497-10510

Liu, Y. X., and Zhao, G. J. (2004). *Wood Science*, China Forestry Publishing House, Beijing, China.

Navi, P., and Girardet, F. (2000). "Effects of thermo-hydro-mechanical treatment on the structure and properties of wood," *Holzforschung* 54(3), 287-293. DOI: 10.1515/HF.2000.048

Navi, P., and Heger, F. (2004). "Combined densification and thermo-hydro-mechanical processing of wood," *MRS Bulletin* 29(5), 332-336. DOI: 10.1557/mrs2004.100

Standfest, G., Kutnar, A., Plank, B., Petutschnigg, A., Kamke, F. A., and Dunky, M. (2013). "Microstructure of viscoelastic thermal compressed (VTC) wood using computed microtomography," *Wood Science and Technology* 47(1), 121-139. DOI: 10.1007/s00226-012-0496-5

Sun, W., Shen, H., and Cao, J. (2016). "Modification of wood by glutaraldehyde and poly (vinyl alcohol)," *Materials and Design* 96, 392-400. DOI: 10.1016/j.matdes.2016.02.044

Welzbacher, C. R., Wehsener, J., Rapp, A. O., and Haller, P. (2008). "Thermo-mechanical densification combined with thermal modification of Norway spruce (*Picea abies* Karst) in industrial scale – Dimensional stability and durability aspects," *Holz als Roh- und Werkstoff* 66(1), 39-49. DOI: 10.1007/s00107-007-0198-0

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